

PATENT
030449

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Examiner: R. T. Shaio :
Art Unit: 1626 :
In re application of:
Zhang, et al. : NEW FLUOROUS TAGGING AND
: SCAVENGING REACTANTS AND
: METHODS OF SYNTHESIS AND
: USE THEREOF
Serial No.: 10/617,431 :
Filed: July 11, 2003 :

**DECLARATION OF ZHIYONG LUO
UNDER 37 CFR § 1.131**

Assistant Commissioner of Patents
Washington, D.C. 20231

Sir:

I, Zhiyong Luo, depose and say:

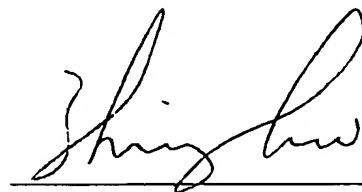
1. I am one of the inventors of the patent application referenced above ("subject application") and of the subject matter described and claimed therein. On information and belief, the Examiner of the subject application has rejected claims 73 through 89 under 35 U.S.C § 102(a) as being unpatentable over the publication by Schwinn et al., "Perfluoro-Tagged Benzyloxycarbonyl Protecting Group and Its Application in Fluorous Biphasic Systems," *Helvetica Chimica Acta*, 2002, 85(1), 255-264, having a publication date of January 2002.

2. Prior to January 2002, I had completed the invention as described and claimed in the subject application in the United States of America as evidenced by the following:

a. Prior to January 2002, after conception of the idea of a fluorous tagging reagent having a structure $X-CR^1R^2-C_6H_{5-m}[W_p(CH_2)_nR_f]_m$, by my co-inventors, Wei Zhang, Tadamichi Nagashima, Christine Hiu-Tung Chen, Marvin S. Yu, and myself, I performed experiments to prepare compounds having structures as set forth in claim 73, 75, and 77, as evidenced by the lab notebook entries on page 71, 73, and 82, attached hereto as Exhibit A. The laboratory notebook pages describe the experimental procedures, quantities of starting materials used, and quantities of products produced. As can be seen from the laboratory notebook pages, compounds having the structure $X-CR^1R^2-C_6H_{5-m}[W_p(CH_2)_nR_f]_m$ were synthesized and used as starting materials in subsequent reaction prior to January 2002. Specifically, the experiment on notebook page 71 shows the synthesis of p-perfluorooctylbenzyl alcohol in 79% yield. The experiments on notebook pages 73 and 82 show experiments where the p-perfluorooctylbenzyl alcohol was used as a reactant in further reactions. Specifically, in the experiment on notebook page 82, p-perfluorooctylbenzyl alcohol was reacted with phosgene and 2-hydroxyimino-2-phenylacetonitrile to form fluorous compound [698].

3. All deleted dates are prior to January 2002.
4. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false

statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issued thereon.

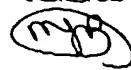


Zhiyong Luo

Aug. 22, 2005

Date

See attached California
All-Purpose Acknowledgment



CALIFORNIA ALL-PURPOSE ACKNOWLEDGMENT

State of California

County of San Diego

ss.

On 8/22/2005 before me, MARIA LOUISA BARBOSA, NOTARY PUBLIC

Date

Name and Title of Officer (e.g., "Jane Doe, Notary Public")

personally appeared Zhiyong Luo

Name(s) of Signer(s)

personally known to me
 proved to me on the basis of satisfactory evidence

to be the person(s) whose name(s) is/are subscribed to the within instrument and acknowledged to me that he/she/they executed the same in his/her/their authorized capacity(ies), and that by his/her/their signature(s) on the instrument the person(s), or the entity upon behalf of which the person(s) acted, executed the instrument.

WITNESS my hand and official seal.

Maria Louisa Barbosa

Signature of Notary Public

Place Notary Seal Above

**OPTIONAL**

Though the information below is not required by law, it may prove valuable to persons relying on the document and could prevent fraudulent removal and reattachment of this form to another document.

Description of Attached DocumentTitle or Type of Document: Declaration/Power 10/611,431Document Date: 8/22/2005Number of Pages: 3

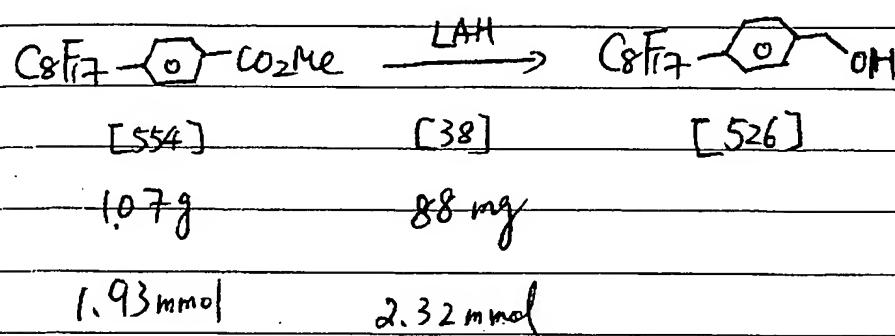
Signer(s) Other Than Named Above: _____

Capacity(ies) Claimed by SignerSigner's Name: ZHIYONG LUO

Individual
 Corporate Officer — Title(s): _____
 Partner — Limited General
 Attorney in Fact
 Trustee
 Guardian or Conservator
 Other: _____

Signer Is Representing: Self

EXHIBIT A



SM, ether, 0°C

Add LAH (powder) at 0°C 9:55 am, 0°C.
0°C for 1.5h

Quench w H₂O

Extraction w/ Et₂O

NMR: pure product

yield: 0.80g (79%)

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Read and Understood By _____

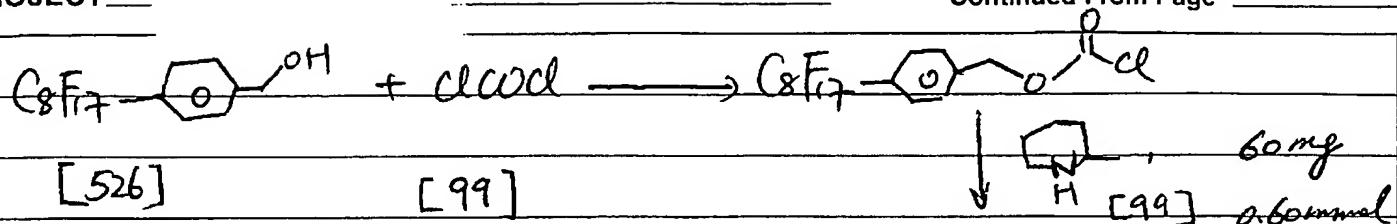
Signed _____

Date _____

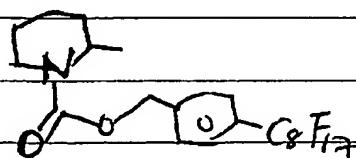
Signed _____

Date _____

PROJECT -



100 mg
0.19 mmol
20% in Tol.
 $d = 0.935$
150 μ L
0.28 mmol



phosgene (20% Soln. in Toluene.) + dry THF (5ml), - ~~0°C~~ 0°C

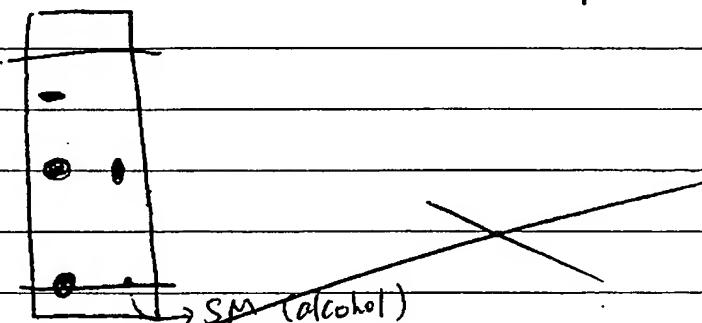
A soln. of alcohol in THF (5 ml) was added dropwise at 0°C for 60'. Evaporated to dryness

Redissolve in 10 ml dry THF.

Add a soln. of CuI in dry THF

R.T. 1:30pm

2:30 pm TLC (30% EtH) workup w/ 1N HCl



No product

Mainly Sm

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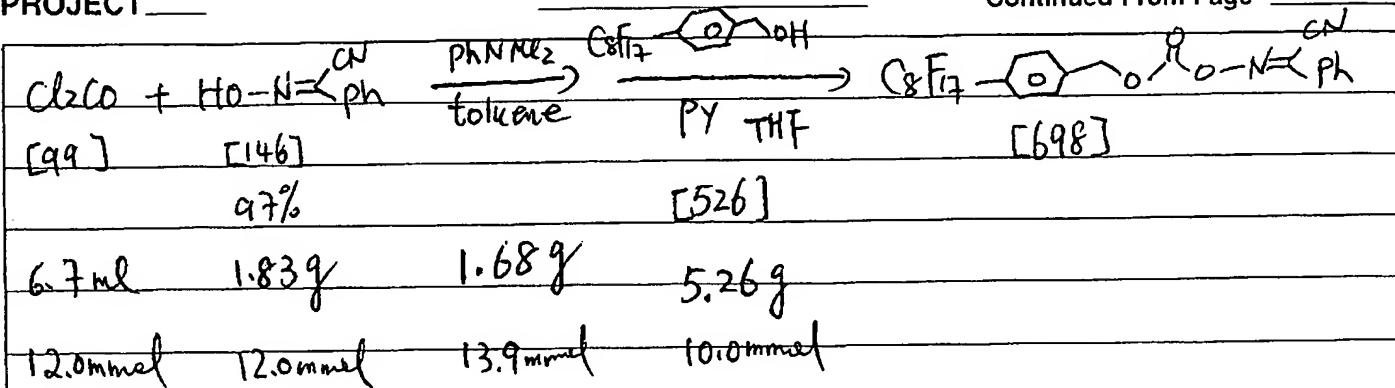
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Date



Phosgene (20% Soln in Tol.) & Toluene (10 ml) at 0°C

Add a soln of $\text{HO}-\text{N}=\text{ph}$ & PhNMe_2 in 1,4-Dioxane (1.0 ml) & toluene (12 ml) at 0°C (2:10 pm) $\xrightarrow{\text{a slurry, heat to } 75^\circ\text{C}}$
 0°C for 2h (4:10 pm) $\xrightarrow{12.5 \text{ mmol})}$

Add a Soln. of alcohol & Py (1.0 ml) in THF (dry, 15 ml) dropwise at $\approx 75^\circ\text{C}$
~~75°C~~ overnight.

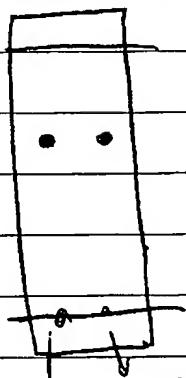
Workup w/ 0.5N HCl

Evaporate solvent

Recrystallized from 100 ml MeOH: pure product

TLC (25% EtOH)

Yield: 5.17 g (74%)



Notice: No benzene

first step, 2h.

2-82 2-77-1A

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Read and Understood By _____

Signed _____

Date _____

Signed _____

Date _____